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Non-Destructive Method for Environmental Breakdown of Graphite Fiber Reinforced Polymer Composites

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AUTHOR(S)

S. Ray Taylor, Leland Melvin, Thomas P. Mangiacapre, George L. Cahen, Jr., and Glenn E. Stoner

PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

University of Virginia
Charlott4sville, VA 22901

8. PERFOR REPORT

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J. S. Army Research Office
P. O. Box 12211
Research Triangle Park, NC 27709-2211

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13. ABSTRACT *(Maximum 200 words)*

Studies were made to determine if moisture ingress down the fiber/ could be detected as an increase in the double layer capacitance C the specific double layer capacitance $C^{n}_{(sp)}$ (muF/cm^2 was determined pitch fibers having an average diameter of 10mu. Individual unsiz electrically contacted to the end of a wire enclosed in a glass pi frame. The free end of this fiber was then secured to the end of with paraffin and $D^{n}_{(dl)}$ measurements were made employing an Electroc technique. By measuring the exposed area of each fiber tested, th be calculated and was found to have an average value of 8.27 ± 9.4

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A model of a CFRP was used to study moisture ingress in these composite systems.

Coating experimentation involved taking ideal electrode, Pt wire, and look at three different coating systems to try and characterize interfacial changes caused by moisture.

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5. NAME OF INSTITUTION: University of Virginia

6. AUTHORS OF REPORT: S. Ray Taylor (Research Assistant Professor)
Leland Melvin (M.S. candidate)
Thomas P. Mangiacapre (M.S. candidate)
George L. Cahen, Jr. (Associate Professor)
Glenn E. Stoner (Professor)

7. LIST OF MANUSCRIPTS SUBMITTED OR PUBLISHED UNDER ARO SPONSORSHIP DURING CONTRACT PERIOD, INCLUDING JOURNAL REFERENCES:

Theses:

D. L. Reichert (MS) - "Electrochemical Measurements for the Evaluation of Composites Containing Simulated In-Service Damage".

R. C. Glass (MS) - "The Use of Electrochemical Characterization Techniques to Nondestructively Evaluate Simulated In-Service Damage in a Carbon Reinforced Plastic".

Journal Articles:

Glass, R. C., Taylor, S. R., Cahen, G. L., Jr. and Stoner, G. E., "Electrochemical Impedance Spectroscopy as a Method to Nondestructively Monitor Simulated In-Service Damage in a Carbon Reinforced Plastic", Journal of Nondestructive Evaluation, Vol. 6, No. 4, 1987, pp 181-188.

Lipka, S. M., Cahen, G. L., Jr., Stoner, G. E., Scribner, L. L., Jr. and Gileadi, E., "The Electrochemical Behavior of Graphite Fiber-Epoxy Composite Electgrodes Containing Varying Fiber Orientations", Vol. 135, No. 2, 1988, pp 368-372.

Lipka, S. M., Cahen, G. L., Jr., Stoner, G. E. and Gileadi, E., "Hydrogen and Oxygen Evolution on Graphite Fiber-Epoxy Matrix Composite Electrodes", Electrochemica Acta, Vol. 33, No. 6, 1988, pp 753-760.

8. SCIENTIFIC PERSONNEL SUPPORTED BY THIS PROJECT AND DEGREES AWARDED DURING THIS CONTRACT PERIOD:

David L. Reichert, M. S. August 1987
Robert C. Glass, M. S. August 1987
Leland Melvin, M. S. candidate (1990)
Thomas P. Mangiacapre, M. S. candidate (1990)
Ray Taylor, Research Assistant Professor
George L. Cahen, Jr., Associate Professor
Glenn E. Stoner, Professor

Glenn E. Stoner
Materials Science
University of Virginia
Charlottesville, VA 22904

Research Findings for Period
January 1, 1989 through December 31, 1989
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Graphite Fiber Experimentation

The purpose of this phase of the research was to determine if moisture ingress down the fiber/epoxy interface could be detected as an increase in the double layer capacitance C_{dl}. To do this, the specific double layer capacitance C_{sp} ($\mu F/cm^2$ was determined for individual pitch fibers having an average diameter of 10μ. Individual unsized fibers were electrically contacted to the end of a wire enclosed in a glass pipette support frame. The free end of this fiber was then secured to the end of the pipette with paraffin and D_{dl} measurements were made employing an Electrochemical Impedance technique. By measuring the exposed area of each fiber tested, the C_{sp} could then be calculated and was found to have an average value of $8.27 \pm 9.4\ \mu F/cm^2$.

A model of a CFRP was used to study moisture ingress in these composite systems. Individual unsized fibers were electrically connected to a wire and set vertically in Epon 828 epoxy. These samples were then cured and polished to 1μ alumina to expose the cross section of the fiber flush with the surface of the epoxy. These samples were then placed in an oven at 90^oC for 10 minutes (causing the interface to debond) and immediately placed in solution for C_{dl} measurements. Upon cooling in solution, air within the interface contracts and draws solution down into the interface. An increase in the C_{dl} was measured over a period of one hour for three samples and corresponded to a final penetration distance of approximately 20μ.

This proves to be a significant capability showing that very small capacitance measurements can be made on individual graphite fibers mounted in a polymer matrix. This leaves work to be done on arrays of fibers mounted in a polymer matrix as the next step in this fundamental study.

Coating Experimentation

The final work on this phase of the project was to take an ideal electrode, Pt wire, and look at three different coating systems to try and characterize interfacial changes caused by moisture. The following three Pt wire electrode configurations were tested. No coating, paraffin coating, and a polyester and paraffin coating. In all cases, no metal-coating interface was exposed to solution as in the graphite fiber work. All moisture ingress would have to diffuse through the polymer in order to get at the interface. Therefore, the impact of moisture diffusion in the coating material would be measured electrochemically. At least three samples from each coating configuration were exposed to 3.5 weight percent sodium sulfate for close to 60 hours. Nyquist plots for the polyester/paraffin coated samples show time constant multiplicity. All Bode angle plots showed depressed circles at early exposure times (0.3 hrs.). At later times (40 hrs.), resolution of the formerly hidden time constants begin to emerge and can be seen from both they Nyquist and Bode plots. A computer program was written to selectively extract phase angle data at a given frequency from each EIS scan taken over the duration of the experiment. Phase angle data for the polyester/paraffin samples show a considerable increase in phase

angle at early times (< 1 hr). Angle data was extracted at 5.163 Hz from each sample. 5.163 Hz was chosen because on all samples, the depressed Bode angle curve had a very similar curvature at this frequency. Therefore, comparisons between angle and time could be made consistently for all samples. Comparisons of angle data show the development or resolution of another time constant from the initial depressed arc. The angle curves for samples with no coating and a paraffin coating changed less than one degree for exposure times less than an hour. However, the polyester/paraffin/ coated sample showed a 5 1/2 to 8 degree change at early times. Sharp increases in capacitance at early times were seen in earlier work done using pencil leads coated with polyester. The polyester/paraffin coated samples show an angle increase of 11-19 degrees over a 60 hour period while the maximum angle increase for either the paraffin coated or bare Pt samples was no more than 8 degrees. Qualitatively, we can say that the addition of a polyester/Pt interface causes different electrochemical activity than in the case of a bare Pt electrode or paraffin coated sample.

Correlation between phase angle and capacitance data is being done as well as trying to tie this work to other work being done in this contract. Comparisons between R.C. Glass' results and these is also being done.

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1 - 50	Army Research Office P. O. Box 12211 Research Triangle Park, N.C. 27709 Attention: Dr. George A. Neece, Acting Director Metallurgy and Materials Science Division
51 - 52	G. Stoner
53	T. Courtney
54 - 55	E. H. Pancake/Clark Hall